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2-(4-Bromophenyl)-*N*-(3-chloro-4-fluorophenyl)acetamide

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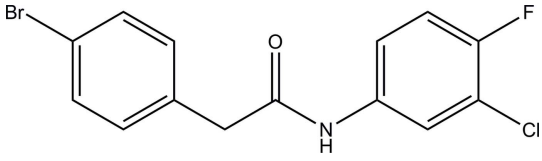
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.006 Å; R factor = 0.054; wR factor = 0.120; data-to-parameter ratio = 27.4.

In the title compound, $C_{14}H_{10}BrClFNO$, the benzene rings form a dihedral angle of 64.0 (2)°. In the crystal, molecules are linked *via* intermolecular N—H···O, C—H···O, C—H···Cl and C—H···F hydrogen bonds into layers parallel to (001). The crystal was refined as a merohedral twin with a 0.935 (114):0.065 (14) domain ratio.

Related literature

For general background to the title compound and for related structures, see: Fun *et al.* (2011*a*,*b*, 2012*a*,*b*). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{14}H_{10}BrClFNO$
M_r = 342.59
Orthorhombic, $P2_12_12_1$
a = 4.9120 (5) Å
b = 6.3131 (6) Å
c = 42.517 (4) Å
V = 1318.4 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.32 mm^{-1}
T = 100 K
0.30 × 0.17 × 0.07 mm

Data collection

Bruker SMART APEXII DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.440, T_{max} = 0.806
10866 measured reflections
4737 independent reflections
4358 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.120
S = 1.15
4737 reflections
173 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.86 e Å^{-3}
$\Delta\rho_{min}$ = −1.82 e Å^{-3}
Absolute structure: Flack (1983), 1929 Friedel pairs
Flack parameter: 0.065 (14)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*1···O1^{i}	0.88	1.97	2.844 (5)	172
C2—H2*A*···O1^{ii}	0.95	2.58	3.321 (5)	135
C10—H10*A*···Cl1^{iii}	0.95	2.67	3.583 (5)	160
C11—H11*A*···F1^{iv}	0.95	2.52	3.443 (6)	165

Symmetry codes: (i) $x+1, y, z$; (ii) $x, y+1, z$; (iii) $x-1, y+1, z$; (iv) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2791).

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§ Thomson Reuters ResearcherID: A-5525-2009.

supplementary materials

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2-(4-Bromophenyl)-*N*-(3-chloro-4-fluorophenyl)acetamide

Hoong-Kun Fun, Ching Kheng Quah, Prakash S. Nayak, B. Narayana and B. K. Sarojini

Comment

In continuation of our work on the synthesis of amides (Fun *et al.*, 2011*a*, 2011*b*, 2012*a*, 2012*b*), we report herein the crystal structure of the title compound.

In the title molecule (Fig. 1), the two benzene rings (C1-C6, C9-C14) form a dihedral angle of 64.0 (2)°. Bond lengths and angles are within normal ranges and are comparable to those found in related structures (Fun *et al.*, 2011*a*, 2011*b*, 2012*a*, 2012*b*). In the crystal structure (Fig. 2), molecules are linked *via* intermolecular N1–H1N1···O1, C2–H2A···O1, C10–H10A···Cl1 and C11–H11A···F1 hydrogen bonds (Table 1) into two-dimensional layers parallel to (001).

Experimental

4-Bromophenylacetic acid (0.213 g, 1 mmol), 3-chloro-4-fluoroaniline (0.145 g, 1 mmol) and 1-ethyl-3-(3-dimethyl-aminopropyl)-carbodiimide hydrochloride (1.0 g, 0.01 mol) were dissolved in dichloromethane (20 mL). The mixture was stirred in presence of triethylamine at 273 K for about 3 h, poured into 100 mL of ice-cold aqueous hydrochloric acid with stirring and was then extracted thrice with dichloromethane. The organic layer was washed with a saturated $NaHCO_3$ solution and brine solution, dried and concentrated under reduced pressure to give the title compound. Single crystals were grown from acetone and toluene (1:1 *v*/*v*) mixture by the slow evaporation method (m.p.: 415-417 K).

Refinement

Atom H1N1 was located in a difference Fourier map and refined using a riding model with U_{iso}(H) = 1.2 U_{eq}(N) [N–H = 0.8825 Å]. The remaining H atoms were positioned geometrically and refined using a riding model with C–H = 0.95 or 0.99 Å and U_{iso}(H) = 1.2 U_{eq}(C). The crystal was refined as an inversion twin with a final refined BASF ratio of 0.935 (114):0.065 (14) for 1929 Friedel pairs.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

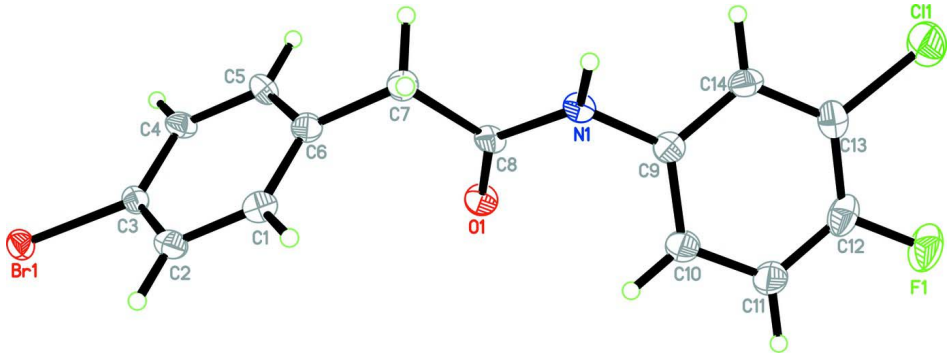

Figure 1

The molecular structure of the title compound showing 50% probability displacement ellipsoids for non-H atoms.

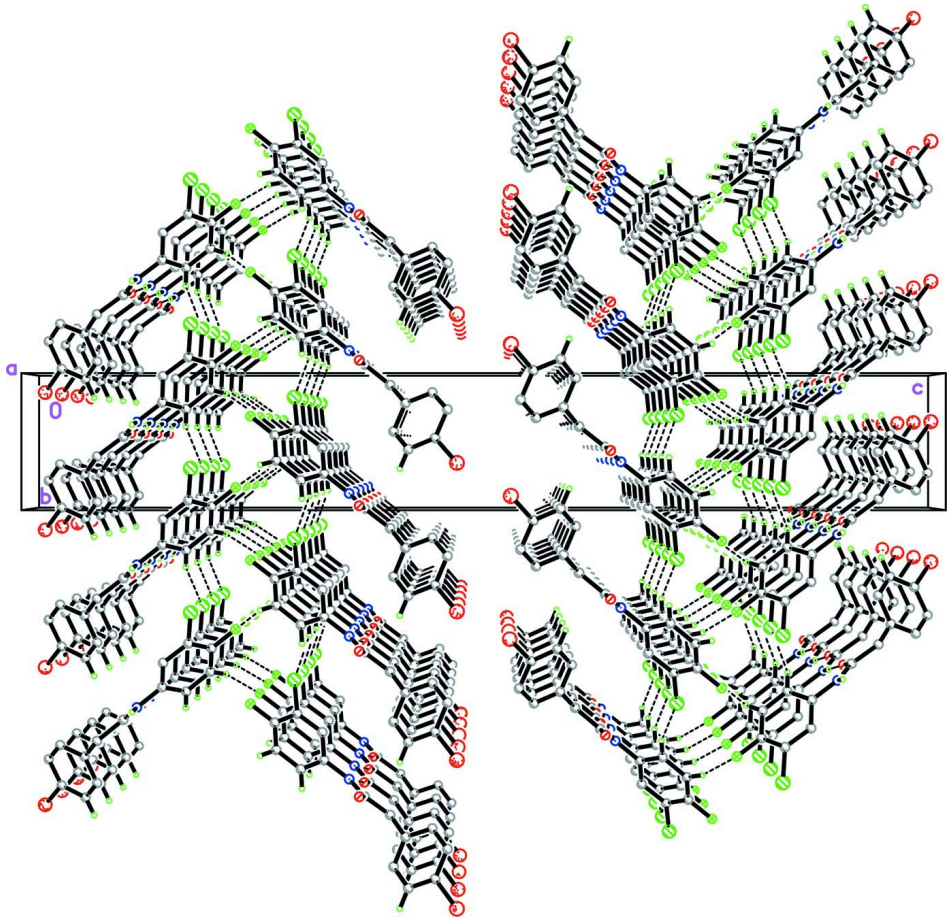

Figure 2

The crystal structure of the title compound, viewed along the *a* axis. H atoms not involved in hydrogen bonds (dashed lines) have been omitted for clarity.

2-(4-Bromophenyl)-*N*-(3-chloro-4-fluorophenyl)acetamide

Crystal data

$C_{14}H_{10}BrClFNO$
$M_r = 342.59$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 4.9120$ (5) Å
$b = 6.3131$ (6) Å
$c = 42.517$ (4) Å
$V = 1318.4$ (2) Å^3
$Z = 4$
$F(000) = 680$
$D_x = 1.726$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4232 reflections
$\theta = 3.5$–$31.8°$
$\mu = 3.32$ mm^{-1}
$T = 100$ K
Plate, colourless
0.30 × 0.17 × 0.07 mm

Data collection

Bruker SMART APEXII DUO CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.440$, $T_{max} = 0.806$
10866 measured reflections
4737 independent reflections
4358 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 32.5°$, $\theta_{min} = 3.3°$
$h = -7 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -46 \rightarrow 63$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.120$
$S = 1.15$
4737 reflections
173 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0038P)^2 + 3.9457P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.86$ e Å^{-3}
$\Delta\rho_{min} = -1.82$ e Å^{-3}
Absolute structure: Flack (1983), 1929 Friedel pairs
Flack parameter: 0.065 (14)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.05032 (9)	1.14272 (6)	0.026647 (9)	0.01959 (9)

Cl1	1.2503 (4)	−0.31406 (18)	0.19870 (3)	0.0379 (3)
F1	0.8185 (7)	−0.1764 (6)	0.24039 (7)	0.0389 (8)
O1	0.5500 (7)	0.4407 (5)	0.12674 (7)	0.0216 (5)
N1	0.9909 (6)	0.3622 (6)	0.13959 (7)	0.0183 (6)
H1N1	1.1678	0.3812	0.1374	0.022*
C1	0.6150 (9)	0.9292 (7)	0.09204 (10)	0.0206 (8)
H1A	0.6939	0.9808	0.1110	0.025*
C2	0.4185 (9)	1.0515 (6)	0.07644 (9)	0.0195 (8)
H2A	0.3622	1.1843	0.0847	0.023*
C3	0.3096 (8)	0.9750 (6)	0.04902 (9)	0.0158 (7)
C4	0.3851 (9)	0.7798 (6)	0.03669 (10)	0.0185 (7)
H4A	0.3046	0.7284	0.0179	0.022*
C5	0.5801 (9)	0.6617 (6)	0.05240 (9)	0.0189 (7)
H5A	0.6354	0.5291	0.0440	0.023*
C6	0.6964 (9)	0.7335 (6)	0.08025 (10)	0.0182 (7)
C7	0.9099 (8)	0.6042 (6)	0.09676 (10)	0.0204 (8)
H7A	1.0466	0.7010	0.1061	0.025*
H7B	1.0043	0.5142	0.0811	0.025*
C8	0.7943 (8)	0.4641 (6)	0.12252 (9)	0.0157 (7)
C9	0.9335 (9)	0.2242 (6)	0.16520 (9)	0.0179 (7)
C10	0.7350 (10)	0.2685 (8)	0.18765 (11)	0.0268 (9)
H10A	0.6262	0.3923	0.1858	0.032*
C11	0.6968 (10)	0.1312 (10)	0.21275 (11)	0.0320 (10)
H11A	0.5588	0.1590	0.2279	0.038*
C12	0.8580 (11)	−0.0440 (8)	0.21570 (11)	0.0275 (9)
C13	1.0540 (12)	−0.0902 (6)	0.19399 (9)	0.0228 (8)
C14	1.0956 (9)	0.0427 (7)	0.16824 (9)	0.0211 (8)
H14A	1.2313	0.0108	0.1530	0.025*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.01688 (15)	0.01907 (14)	0.02281 (16)	0.00264 (16)	0.00107 (16)	0.00387 (15)
Cl1	0.0564 (9)	0.0216 (5)	0.0357 (6)	0.0089 (5)	−0.0016 (6)	−0.0003 (4)
F1	0.0354 (18)	0.0496 (19)	0.0316 (14)	−0.0056 (16)	0.0028 (13)	0.0207 (14)
O1	0.0082 (11)	0.0314 (13)	0.0253 (13)	−0.0002 (14)	0.0008 (13)	0.0045 (11)
N1	0.0022 (14)	0.0290 (14)	0.0236 (14)	−0.0016 (13)	0.0004 (10)	0.0048 (14)
C1	0.020 (2)	0.0221 (16)	0.0193 (17)	−0.0013 (15)	0.0019 (15)	−0.0015 (14)
C2	0.017 (2)	0.0183 (15)	0.0229 (17)	0.0020 (16)	0.0024 (16)	−0.0017 (13)
C3	0.0116 (17)	0.0151 (14)	0.0206 (17)	−0.0001 (13)	0.0018 (14)	0.0030 (12)
C4	0.0139 (18)	0.0213 (16)	0.0202 (16)	0.0035 (14)	−0.0002 (14)	−0.0024 (13)
C5	0.0202 (19)	0.0135 (14)	0.0231 (16)	0.0030 (16)	−0.0007 (15)	−0.0006 (13)
C6	0.0142 (18)	0.0204 (16)	0.0200 (18)	0.0003 (15)	0.0013 (15)	0.0015 (14)
C7	0.0114 (19)	0.0263 (19)	0.0235 (17)	0.0004 (14)	0.0028 (14)	0.0065 (14)
C8	0.0067 (15)	0.0216 (16)	0.0187 (17)	−0.0004 (14)	−0.0027 (13)	−0.0020 (13)
C9	0.0082 (15)	0.0261 (16)	0.0194 (16)	−0.0035 (16)	−0.0021 (16)	0.0007 (13)
C10	0.018 (2)	0.038 (2)	0.025 (2)	0.0064 (19)	0.0039 (17)	0.0044 (17)
C11	0.019 (2)	0.051 (3)	0.026 (2)	0.009 (2)	0.0075 (17)	0.011 (2)
C12	0.027 (2)	0.033 (2)	0.023 (2)	−0.007 (2)	−0.0017 (18)	0.0093 (17)
C13	0.027 (2)	0.0179 (15)	0.0234 (17)	−0.0008 (18)	−0.0069 (19)	−0.0003 (12)

C14	0.018 (2)	0.0259 (18)	0.0190 (17)	0.0000 (16)	0.0040 (15)	−0.0017 (14)

Geometric parameters (Å, º)

Br1—C3	1.910 (4)	C5—C6	1.391 (6)
Cl1—C13	1.723 (4)	C5—H5A	0.9500
F1—C12	1.356 (5)	C6—C7	1.503 (6)
O1—C8	1.222 (5)	C7—C8	1.518 (6)
N1—C8	1.369 (5)	C7—H7A	0.9900
N1—C9	1.423 (5)	C7—H7B	0.9900
N1—H1N1	0.8825	C9—C10	1.393 (6)
C1—C6	1.392 (6)	C9—C14	1.401 (6)
C1—C2	1.403 (6)	C10—C11	1.388 (7)
C1—H1A	0.9500	C10—H10A	0.9500
C2—C3	1.371 (6)	C11—C12	1.366 (7)
C2—H2A	0.9500	C11—H11A	0.9500
C3—C4	1.390 (5)	C12—C13	1.365 (7)
C4—C5	1.385 (6)	C13—C14	1.395 (6)
C4—H4A	0.9500	C14—H14A	0.9500
C8—N1—C9	123.6 (3)	C6—C7—H7B	109.0
C8—N1—H1N1	125.0	C8—C7—H7B	109.0
C9—N1—H1N1	111.2	H7A—C7—H7B	107.8
C6—C1—C2	121.1 (4)	O1—C8—N1	123.9 (4)
C6—C1—H1A	119.5	O1—C8—C7	122.9 (4)
C2—C1—H1A	119.5	N1—C8—C7	113.1 (3)
C3—C2—C1	118.5 (4)	C10—C9—C14	119.9 (4)
C3—C2—H2A	120.8	C10—C9—N1	122.7 (4)
C1—C2—H2A	120.8	C14—C9—N1	117.3 (4)
C2—C3—C4	122.0 (4)	C11—C10—C9	119.8 (4)
C2—C3—Br1	119.2 (3)	C11—C10—H10A	120.1
C4—C3—Br1	118.8 (3)	C9—C10—H10A	120.1
C5—C4—C3	118.7 (4)	C12—C11—C10	119.9 (4)
C5—C4—H4A	120.7	C12—C11—H11A	120.1
C3—C4—H4A	120.7	C10—C11—H11A	120.1
C4—C5—C6	121.3 (4)	F1—C12—C13	119.5 (4)
C4—C5—H5A	119.4	F1—C12—C11	119.2 (5)
C6—C5—H5A	119.4	C13—C12—C11	121.3 (4)
C5—C6—C1	118.6 (4)	C12—C13—C14	120.4 (4)
C5—C6—C7	120.5 (4)	C12—C13—Cl1	119.4 (3)
C1—C6—C7	120.9 (4)	C14—C13—Cl1	120.2 (4)
C6—C7—C8	113.1 (3)	C13—C14—C9	118.7 (4)
C6—C7—H7A	109.0	C13—C14—H14A	120.6
C8—C7—H7A	109.0	C9—C14—H14A	120.6
C6—C1—C2—C3	−0.7 (6)	C8—N1—C9—C10	−41.6 (6)
C1—C2—C3—C4	1.1 (6)	C8—N1—C9—C14	141.6 (4)
C1—C2—C3—Br1	−178.0 (3)	C14—C9—C10—C11	−0.7 (7)
C2—C3—C4—C5	−1.2 (6)	N1—C9—C10—C11	−177.4 (4)
Br1—C3—C4—C5	177.8 (3)	C9—C10—C11—C12	1.5 (8)

C3—C4—C5—C6	1.1 (6)	C10—C11—C12—F1	179.8 (5)
C4—C5—C6—C1	−0.7 (6)	C10—C11—C12—C13	−1.4 (8)
C4—C5—C6—C7	−179.3 (4)	F1—C12—C13—C14	179.3 (4)
C2—C1—C6—C5	0.5 (6)	C11—C12—C13—C14	0.5 (8)
C2—C1—C6—C7	179.1 (4)	F1—C12—C13—Cl1	−0.8 (6)
C5—C6—C7—C8	−95.2 (5)	C11—C12—C13—Cl1	−179.6 (4)
C1—C6—C7—C8	86.3 (5)	C12—C13—C14—C9	0.2 (7)
C9—N1—C8—O1	−2.9 (6)	Cl1—C13—C14—C9	−179.6 (3)
C9—N1—C8—C7	179.2 (4)	C10—C9—C14—C13	−0.1 (6)
C6—C7—C8—O1	8.2 (6)	N1—C9—C14—C13	176.7 (4)
C6—C7—C8—N1	−174.0 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*1···O1^{i}	0.88	1.97	2.844 (5)	172
C2—H2*A*···O1^{ii}	0.95	2.58	3.321 (5)	135
C10—H10*A*···Cl1^{iii}	0.95	2.67	3.583 (5)	160
C11—H11*A*···F1^{iv}	0.95	2.52	3.443 (6)	165

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*, *y*+1, *z*; (iii) *x*−1, *y*+1, *z*; (iv) −*x*+1, *y*+1/2, −*z*+1/2.